02-08-00

A

jc772 U.S. PTO 02/07/00

EXPRESS MAIL CERTIFICATE

Date 2/7/2000 Label No. EL503340130

I hereby certify that, on the date indicated above I deposited this paper or fee with the U.S. Postal Service & that it was addressed for delivery to the Assistant Commissioner of Patents & Trademarks, Washington, DC 20231 by "Express Mail Post Office to Addressee" service.

D B Peek D B Peek

Name (Print) Signature

jc530 U.S. PTO 09/499207 02/07/00

PLEASE CHARGE ANY DEFICIENCY UP TO $300.00 OR CREDIT ANY EXCESS IN THE FEES DUE WITH THIS DOCUMENT TO OUR DEPOSIT ACCOUNT NO. 04-0100

DARBY & DARBY P.C.
805 Third Avenue
New York, New York 10022
212-527-7700

Docket No: 2136/0G684

Hon. Commissioner of
Patents and Trademarks
Washington, DC 20231

Sir:

Enclosed please find an application for United States patent as identified below:

Inventor/s (name ALL inventors): Jose Oriol **GUIXA ARDERIU;** Miquel **GARCIA ZAMORA**; Ferran **ESPIELL ALVAREZ**; Miquel Angel **FERNANDEZ LOPEZ;** Araceli **ESPARDUCER BROCO**; Merce **SEGARRA RUBIK**; and Josep Ma **CHIMENOS RIBERA**

Title: **MANUFACTURE OF COPPER MICROALLOYS**

including the items indicated:

1. Specification and 4 claims: 1 indep.; 3 dep.; 0 multiple dep.

2. [**X**] Executed declaration and power of attorney
[] Unexecuted declaration and power of attorney

3. [] Formal drawings, _ sheets (Figs.)
[X] Informal drawings, 1 sheet (Fig. 1)

4. [X] Assignment for recording to: LA FARGA LACAMBRA, S.A.

5. [X] Verified Statement Claiming Small Entity Status

6. [X] Check in amount of $385.00, ($345.00 filing; $40.00 recording; $ surcharge)
(See attached **Fee Computation Sheet**)

7. [X] Preliminary Amendment.

8. [X] Please amend the description by inserting the following paragraph after the line containing the title on page 1:
"This patent application claims priority under 35 U.S.C. § 119 from Spain Patent Application No. 9900254, filed February 8, 1999."

Priority is claimed for this application under 35 U.S.C. § 119, corresponding application/s having been filed as follows:

Country:	Spain
Number:	9900254
Date:	February 8, 1999

The priority documents [] are enclosed
[X] will follow.

February 7, 2000

Respectfully submitted,

Paul Fields, Esq.
Reg. No. 20,298
Attorney for Applicants
DARBY & DARBY P.C.
805 THIRD AVENUE
NEW YORK, NEW YORK 10022
(212) 527-7700

PATENT FEE COMPUTATION SHEET

	No. of Claims Presented	Extra Claims Previously Paid For	Number of Extra Claims	Rate
Basic Fee				$690.00
Total Claims	4 - 20	- 0 = 0	x $18.00	$0.00
Independent Claims	1 - 3	- 0 = 0	x $78.00	$0.00
Multiple Dependent Claims		-if so, add $260.00		$.00
Surcharge for late submission of filing fee and/or declaration ($130.00)				$0.00
SUBTOTAL				$690.00
[X] Small Entity REDUCTION (Half of Subtotal)				$345.00
Fee for recordation of assignment ($40.00)				$40.00
Charge for filing non-English language application ($130.00)				$0.00
TOTAL				$385.00

M:\2136\0G684\PAR1018 WPD

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application or Patent of: Docket No.

Serial or Patent No: Filed: or Issued:

For: MANUFACTURE OF COPPER MICROALLOYS

VERIFIED STATEMENT CLAIMING SMALL ENTITY STATUS
SMALL BUSINESS CONCERN

I hereby declare that I am

[ ] the owner of the small business concern identified below:
[X] an official of the small business concern empowered to act on behalf of the concern identified below:

NAME OF CONCERN:
ADDRESS OF CONCERN:

I hereby declare that the above identified small business concern qualifies as a small business concern as defined in 13 CFR 121.12 and in 37 CFR 1.9(d), for purposes of paying reduced fees to the United States Patent and Trademark Office, in that the number of employees of the concern, including those of its affiliates, does not exceed 500 persons.

Definitions: For purposes of this statement, (1) the number of employees of the business concern is the average over the previous fiscal year of the concern of the persons employed on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when either, directly or indirectly, one concern controls or has the power to control the other, or a third party or parties controls or has the power to control both.

I hereby declare that rights under contract or law have been conveyed to and remain with the small business concern identified above with regard to the invention entitled _____ _____, by inventor(s) _____ described in

[X] the specification filed herewith
[ ] application Serial No. _____, filed _____
[ ] Patent No. _____, issued _____.

If the rights held by the above identified small business concern are not exclusive, each individual, concern or organization having rights to the invention is listed below* and no

rights to the invention are held by any person, other than the inventor, who could not qualify as an independent inventor under 37 CFR 1.9(c) if that person made the invention, or by any concern which would not qualify as a small business concern under 37 CFR 1.9(d), or a nonprofit organization under 37 CFR 1.9(e).

*NOTE: *Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entitled (37 C.F.R. 1.27)*

NAME:

ADDRESS:

[ ] INDIVIDUAL [ ] SMALL BUSINESS CONCERN [ ] NONPROFIT ORGANIZATION

NAME:

ADDRESS:

[ ] INDIVIDUAL [ ] SMALL BUSINESS CONCERN [ ] NONPROFIT ORGANIZATION

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 C.F.R. §1.28(b))

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statement and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

NAME OF PERSON SIGNING: ORIOL QUIXÀ ARDERIU
TITLE OF PERSON (IF OTHER THAN OWNER): GENERAL MANAGER
ADDRESS OF PERSON SIGNING: Cardenal Reig nº 13 BARCELONA SPAIN

SIGNATURE: _____ DATE: 18. 01. 2000

EXPRESS MAIL CERTIFICATE

Date 2/7/2000 Label No. EL503340130

I hereby certify that, on the date indicated above I deposited this paper or fee with the U.S. Postal Service & that it was addressed for delivery to the Assistant Commissioner of Patents & Trademarks, Washington, DC 20231 by "Express Mail Post Office to Addressee" service.

D B Peck L Beck

Name (Print) Signature

PLEASE CHARGE ANY DEFICIENCY UP TO $300.00 OR CREDIT ANY EXCESS IN THE FEES DUE WITH THIS DOCUMENT TO OUR DEPOSIT ACCOUNT NO. 04-0100

Docket No: 2136/0G684

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Jose Oriol **GUIXA ARDERIU**; Miquel **GARCIA ZAMORA**; Ferran **ESPIELL ALVAREZ**; Miquel **Angel FERNANDEZ LOPEZ** et al.

Serial No.: To Be Assigned Art Unit: To Be Assigned

Filed: Herewith Examiner: To Be Assigned

For: **MANUFACTURE OF COPPER MICROALLOYS**

PRELIMINARY AMENDMENT

Hon. Commissioner of
Patents and Trademarks
Washington, DC 20231

February 7, 2000

Sir:

Prior to examination, please amend the specification by adding after the title:

--This patent application claims priority under 35 U.S.C. § 119 from Spain Patent Application No. 9900254, filed February 8, 1999--.

IN THE CLAIMS:

Claim 3, line 1, delete "and 2";

Claim 3, line 8, delete "and 2".

Claim 4, line 5, delete "and 2".

REMARKS

Allowance of this application is respectfully requested.

Respectfully submitted,

Paul Fields, Esq.
Reg. No. 20,298
Attorney for Applicants

DARBY & DARBY, P.C.
805 Third Avenue
New York, N.Y. 10022
Phone (212) 527-7700

M:\2136\0G684\PAR1024 WPD

MANUFACTURE OF COPPER MICROALLOYS

DESCRIPTION

FIELD OF THE INVENTION

The present invention relates to the manufacture of copper microalloys, particularly the casting copper by conventional batch casting, semi-continuous casting or continuous casting and of rolling tough-pitch copper or microalloyed copper. It provides the addition of lead or refining to a final concentration of lead equal to or higher than 200 ppm. This allows the casting of copper microalloyed with elements such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te in amounts of the order of tens of weight ppm.

This invention also relates to a pre-heating treatment which has been discovered to be necessary to let some copper microalloys with a lead concentration equal to or higher than 200 ppm have the same strain strength, annealing temperature, half-softening temperature and recrystallization temperature as those obtained for tough-pitch copper, and an electrical conductivity equal to or higher than 101.5%IACS.

BACKGROUND OF THE INVENTION

Until recently, it was accepted that a lead content in melt copper higher than 15-20 weight ppm, and a high content of other impurities was undesirable due to reduction of the electrical conductivity and the formation of high number of defects and bubbles in a phenomena known as hot-shortness. This meant that only tough-pitch copper could be cast. Thus blister copper or copper scrap refined by pyrometallurgical methods, which gave a lead content lower than 15-20 weight ppm, and decreased sufficiently impurities to produce high electrical conductivity copper, was not technologically competitive compared to electrolytically-refined copper.

Despite all the related handicaps in the fire-refining process and in the products, some companies developed different slagging agents in order to achieve the purity of tough-pitch copper while avoiding the expensive process of electrolytical refining. Nevertheless, it was difficult to decrease lead content to values lower than 15-20 weight ppm by fire-refining. The fire-refined copper produced was a high quality product, with electrical, thermal and mechanical properties very similar to electrolytically-refined copper, but because of its high lead content, it was often impossible to cast or roll, or else the final product was brittle and susceptible to breakage due to the porosity in the metal.

It is also known that a pre-heating treatment at 550-650°C for one hour or longer before high cold-working (80% or more) on microalloys with a lead content higher than 15-20 ppm significantly decreases their annealing temperature, half-softening temperature and recrystallization temperature, and also increases the electrical conductivity. Some compositions of microalloyed copper, treated with this pre-heating process achieve similar mechanical, thermal and electrical properties to tough-pitch copper.

OBJECT OF THE INVENTION

To find a solution to the above mentioned drawbacks, the inventors have carried out investigations which have led to this invention, providing in a process for the discontinuous, semi-continuous or continuous casting of copper or microalloyed copper, the addition of lead or refining to a final concentration of lead equal to or higher than 200 weight ppm. Surprisingly enough, this allows the casting and rolling of microalloys with impurities such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te in concentrations of the order of tens of weight

ppm.

The invention refers as well to an optional pre-heating treatment of 5-600s which, when applied to some copper microalloys with a lead content equal to or higher than 200 weight ppm, leads to a decrese in their strain strength, giving annealing temperatures, half-softening temperatures or recrystallization temperatures equal to or lower than 200°C to allow the obtention of mechanical, thermal and electric properties similar to ETP-CU.

SUMMARY OF THE INVENTION

This invention is based on the following:

a) Lead concentrations higher than 200 weight ppm in copper and copper microalloys secure their castability by conventional casting (by batch, semi-continuous or continuous casting) and their rolling because of their low hot-shortness, and the number of breaks in the cast bar decreases. The improvement in the microstructure in terms of small number of voids and bubbles also ensures small number of breaks at lower values of tensile strength and elongation than the statistically established.

b) Lead concentrations higher than 200 weight ppm secures the casting and rolling of copper microalloys containing microalloying elements such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te in the order of tens of weight ppm.

c) A pre-heating treatment at 550-650°C for 5-600s on copper with impurity contents lower than 80 weight ppm of elements Sn, Zn, Ni, Ag, Cd, Sb, S and Fe that have been casted by the addition of lead or refining until a lead content in the solid product higher than 300 weight ppm decreases their half-softening temperature, annealing temperature and recrystallization temperature to values lower than 200°C.

DESCRIPTION OF THE DRAWINGS

Fig. 1 depicts the variation in the Softening temperature with the pre-heating time at 586°C in sample 1.

DETAILED DESCRIPTION

It is known that for a lead content in copper lower than 15-20 weight ppm and an oxygen content between 60-400 weight ppm, casting and rolling problems caused by the high hot-shortness are not usual, and the final product has a low number of voids and bubbles. Impurities such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te at less concentration than 5-10 weight ppm are also desirable in order to secure the casting and rolling of copper.

In contrast, copper with more than 15-20 weight ppm of lead and/or other impurities such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te in contents in the order of tens of weight ppm has casting and rolling problems and its microstructure has high number of defects which are large enough to break the cast bar during casting. This is one of the reasons why most of the copper smelters and refiners have adopted the electrolytically-refining method. The formation of casting bubbles and defects can be attributed to the elimination of hydrogen. Hydrogen is formed in the reduction reaction, in which methane is burnt in the reduction furnace. As the lead content increases, the amount of solved hydrogen also increases, reaching the maximum content in hydrogen that in those conditions can be solved in copper when the lead content is between 15-20 and 200 weight ppm. When this copper microalloy is being cast, the excess solved hydrogen is eliminated by the formation of bubbles and voids that can break the casting bar.

The experiences which have been carried out show surprising results because for a lead content in copper

higher than 200 weight ppm, the number of bubbles and voids formed during casting decreases markedly, allowing the casting and rolling of this copper. This may be because the atomic volume of lead is greater than that of copper, because the substitution of copper by lead in the crystal net creates interstices that can be occupied by hydrogen.

Another surprising result is that a lead content in copper higher than 200 weight ppm secures or even improves the castability and rolling of copper microalloys with impurities such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te when their contents are of the order of tens weight ppm, in comparison with the castability of copper with less than 15 ppm of lead. A comparison between the microstructure of casting bars with a lead content lower than 15-20 weight ppm and others with high amounts of microalloying elements and a lead content higher than 300 weight ppm, shows a low number of bubbles and defects in the latter.

However, because of the high lead content, copper microalloys obtained by this casting method have higher strain strengths, higher recrystallization temperatures, higher softening temperatures and higher annealing temperatures than those obtained from the copper or copper microalloys with equivalent composition but a lead content lower than 15-20 weight ppm.

The invention, in order to improve the above mentioned aspect introduces an optional thermal treatment at 550-650°C known as pre-heating. This treatment decreases the softening temperature, annealing temperature and recrystallization temperature in copper and copper microalloys when the lead content is higher than 15-20 weight ppm. This phenomenon is related to the presence of hydrogen in copper and copper microalloys, because pre-

heating decreases the hydrogen and oxygen content. The hydrogen content is around 0.5-0.7 weight ppm after casting by this method, and decreases until there is no detectable hydrogen after a complete pre-heating, when the softening temperature, annealing temperature and recrystallization temperature reach their minimum value, normally after long pre-heating times (2 hours or more).

Another surprising result of the invention is that some coppers, cast after securing a lead content higher than 300 weight ppm, have fast pre-heating kinetics, with a decrease in softening temperature, annealing temperature and recrystallization temperature by a maximum of 30°C in 10 minutes and more specifically, achieving softening temperatures, annealing temperatures and recrystallization temperatures lower than or around 200°C after 5-600s of pre-heating. These coppers need a content lower than 80 weight ppm of other elements such as Sn, Zn, Sb, Cd, Ni, Fe, Bi and S, but a high lead content, always higher than 300 weight ppm and preferably higher than 350 weight ppm. Pre-heating removes hydrogen from the interstitial positions next to lead, forming water, which explains the decrease in oxygen content observed after pre-heating. A low content of microalloying elements which have a higher affinity for oxygen than copper, such as those described before, assists the formation of water, improving the pre-heating kinetics.

The major advantages of this invention are as follows:

i) A lead content higher than 200 weight ppm secures the casting and rolling of copper and copper microalloys, even with impurities in copper microalloys of tens of weight ppm of elements such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te.

ii) The optional pre-heating treatment proposed often

increases the electrical conductivity of the described copper microalloys in comparison with the equivalent coppers of the same composition but with lead contents lower than 15-20 weight ppm.

EXAMPLES

Table 1 shows coppers and copper microalloys produced by semi-continuous casting in an industrial plant by the method proposed, starting from copper scrap that had been fire-refined. Copper microalloys with an Sb content of 20 weight ppm or more and a S content between 3 and 12 weight ppm were cast and rolled with low hot-shortness. Table 2 shows the softening temperatures (defined as the temperature at which the strain strength starts decreasing after of 80% cold-working) of the coppers and copper microalloys described in table 1.

Sample 1 is a copper microalloy as described above which showed a rapid pre-heating. Fig. 1 shows that in 10s of pre-heating, the softening temperature decreased from 192°C to 178°C, reaching 155°C after 600s.

ADVANTAGES OF THE INVENTION

As described above, the present invention provides a new method for casting and rolling copper and copper microalloys, even with microalloying elements such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te in amounts in the order of tens of weight ppm, by batch, semi-continuous or continuous casting that reduces the residence time and energy costs of a fire-refining copper production plant, and gives a casting product with low hot-shortness which has low number of failures in service. Despite the small increase in the softening temperature, annealing temperature and recrystallization temperature, and the decrease in the electrical conductivity of the product produced by this method, compared with the equivalent copper or copper microalloy of the same composition but

with a lead content lower than 15-20 weight ppm, the use of a fast and economical heat treatment, known as pre-heating, decreases the softening temperature, annealing temperature and recrystallization temperature of some copper compositions casted by the proposed method to values less than or around 200°C and leads to the electrical conductivity of tough-pitch copper or even higher

Table 1

Examples of some copper compositions and copper microalloys cast by this method (in weight ppm)

Sample	Pb	Sn	Ni	Ag	Cd	Bi	Sb	Fe	Zn	S	Oxygen
1	479	65	25	28	0.3	0.9	11	11	39	3	168
2	460	23	14	18	0.2	0.6	20	21	15	6	163
3	322	11	9	9	0.8	0.8	5	5	6	5	178
4	520	50	32	19	0.9	0.8	15	14	23	12	218
5	345	46	34	23	1.1	1.0	21	28	24	9	195
6	247	50	30	43	0.9	1.2	22	34	14	6	171
7	236	121	106	59	0.8	0.7	17	27	57	6	154
8	341	81	61	52	0.8	0.6	18	29	80	7	148
9	388	74	69	70	0.6	0.7	22	26	81	10	150

Table 2

Sample	Softening temperature (°C)
1	192
2	192
3	198
4	200
5	210
6	222
7	230
8	242
9	242

Although the vention has been explained based on the aforegoing description and examples it is obvious that the men of the art will find variations which will be included in the field covered by the invention provided that they do not depart from the enclosed claims.

CLAIMS

What we claim is:

1. A method for the manufacture of copper microalloys characterized by starting from a copper alloy containing impurities such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te in amounts of the order of tens of weight ppm, comprising the addition of lead to a final concentration of 200 weight ppm or higher in the solid microalloy or of refining the copper microalloy to said concentration.

2. The method according to claim 1, applied to batch casting, semi-continuous casting or continuous casting.

3. The method according to claims 1 and 2, characterized by optionally comprising a pre-heating treatment at 550-650°C for 5-600 s that decreases the softening temperature, the annealing temperature and the recrystallization temperature to values of 200°C or lower, of copper microalloys with less than 80 weight ppm of the impurities Zn, Ag, Cd, Sb, Ni, Fe, Bi, Sn and S, produced by the casting method described in claims 1 and 2.

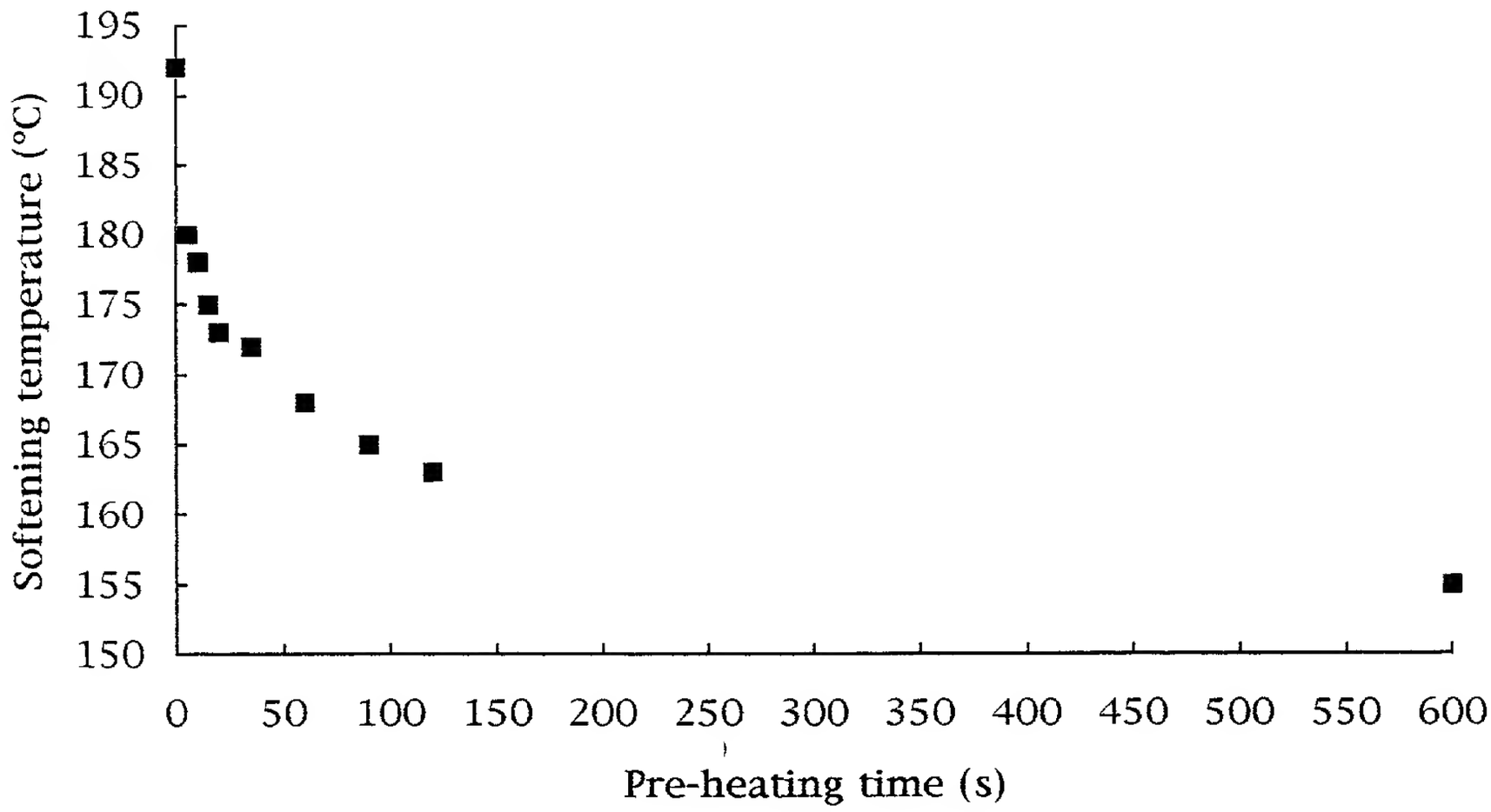
4. A pre-heating treatment at 550-650°C for 5-600 s that, applied to the copper microalloy with contents lower than 80 weight ppm of the impurities Zn, Ag, Cd, Sb, Ni, Fe, Bi, Sn and S, produced by the casting method described in claims 1 and 2, increases the electrical conductivity to the values of the tough-pitch copper, which means 101%IACS or more.

ABSTRACT

The invention refers to batch casting, semi-continuous casting or continuous casting and rolling of copper, providing the addition of lead or refining the melt copper or the melt microalloyed copper to a lead content equal to or higher than 200 weight ppm. This minimizes the number of pores and defects, decreasing the number of incidences or breaks during casting and in service. However, it does not reduce the electrical conductivity. The addition of lead allows the cast and roll of copper microalloyed with elements such as S, Se, As, Sb, Bi, Sn, Zn, Ni, Fe, Ag and Te, in concentrations of the order of tens of weight ppm. The copper microalloys manufactured in this way have annealing temperatures and strain strengths higher than those obtained from the equivalent tough-pitch copper or the equivalent microalloyed copper with lead content lower than 15-20 weight ppm.

This patent also includes a heat treatment a 550-650°C for 5-600s that, when applied to some compositions of the microalloys resulting of the related casting method gives similar values of annealing temperature, half-softening temperature, recrystallization temperature and strain-strength as tough-pitch copper. In addition, electrical conductivity is increased to 101.5%IACS or even higher.

FIG. 1


Softening temperature (°C)
195
190
185
180
175
170
165
160
155
150
0
50
100
150
200
250
300
350
400
450
500
550
600
Pre-heating time (s)

FILE NO.:

DECLARATION
AND POWER OF ATTORNEY
Original Application

As a below named inventor, I declare that the information given herein is true, that I believe that I am the original, first and sole inventor if only one name is listed at 1 below, or a joint inventor if plural inventors are named below, of the invention entitled: MANUFACTURE OF COPPER MICROALLOYS

which is described and claimed in:

[X] the attached specification or [ ] the specification in application Serial No. , filed
(for declaration not accompanying appl.)

that I do not know and do not believe that the same was ever known or used in the United States of America before my or our invention thereof or patented or described in any printed publication in any country before my or our invention thereof, or more than one year prior to this application, or in public use or on sale in the United States of America more than one year prior to this application, that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representatives or assigns more than twelve months prior to this application, that I acknowledge my duty to disclose information of which I am aware which is material to patentability in accordance with 37 CFR §1.56. I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above. I hereby claim the priority benefits under 35 U.S.C. 119 of any application(s) for patent or inventor's certificate listed below. All foreign applications for patent or inventor's certificate on this invention filed by me or my legal representatives or assigns prior to the application(s) of which priority is claimed are also identified below.

PRIOR APPLICATION(S), IF ANY, OF WHICH PRIORITY IS CLAIMED

COUNTRY	APPLICATION NO.	DATE OF FILING
SPAIN	PATENT APPLICATION Nº 9900254	8TH FEBRUARY 1999

ALL FOREIGN APPLICATIONS, IF ANY, FILED PRIOR TO THE APPLICATION(S) OF WHICH PRIORITY IS CLAIMED

COUNTRY	APPLICATION NO.	DATE OF FILING

POWER OF ATTORNEY:

As a named inventor, I hereby appoint the following attorney(s) and/or agents(s) to prosecute this application and transact all business in the Patent and Trademark office connected therewith: Gordon D. Coplein #19,165, William F. Dudine, Jr. #20,569, Michael J. Sweedler #19,937, S. Peter Ludwig #25,351, Paul Fields #20,298, Harold E. Wurst #22,183, Joseph B. Lerch #26,936, Melvin C. Garner #26,272, Ethan Horwitz #27,646, Beverly B. Goodwin #28,417, Adda C. Gogoris #29,714, Martin E. Goldstein #20,869, Bert J. Lewen #19,407, Henry Sternberg #22,408, Robert A. Green #28,301, Peter C. Schechter #31,662, Robert Schaffer #31,194, David R. Francescani #25,159, Robert C. Sullivan, Jr. #30,499, Ira J. Levy #35,587, Joseph R. Robinson #33,448

all of the firm of DARBY & DARBY P.C., 805 Third Avenue, New York, NY 10022

SEND CORRESPONDENCE TO:

DARBY & DARBY P.C.
805 Third Avenue
New York, NY 10022

DIRECT TELEPHONE CALLS TO:

PAUL FIELDS

212-527-7700

FULL NAME AND RESIDENCE OF INVENTOR 1

LAST NAME: GUIXÀ ARDERIU FIRST NAME: José Oriol MIDDLE NAME:
CITY: BARCELONA STATE OR FOREIGN COUNTRY: SPAIN COUNTRY OF CITIZENSHIP: SPAIN
POST OFFICE ADDRESS: Cardenal Reig, 13 CITY: Barcelona STATE OR COUNTRY: SPAIN ZIP CODE: 08028

FULL NAME AND RESIDENCE OF INVENTOR 2

LAST NAME: GARCIA ZAMORA FIRST NAME: Miquel MIDDLE NAME:
CITY: MOIÀ (Barcelona) STATE OR FOREIGN COUNTRY: SPAIN COUNTRY OF CITIZENSHIP: SPAIN
POST OFFICE ADDRESS: Tramuntana, 14 CITY: Moià Barcelona STATE OR COUNTRY: SPAIN ZIP CODE: 08180

FULL NAME AND RESIDENCE OF INVENTOR 3

LAST NAME: ESPIELL ÀLVAREZ FIRST NAME: Ferran MIDDLE NAME:
CITY: BARCELONA STATE OR FOREIGN COUNTRY: SPAIN COUNTRY OF CITIZENSHIP: SPAIN
POST OFFICE ADDRESS: Gran Vía Carles III, 50 CITY: Barcelona STATE OR COUNTRY: SPAIN ZIP CODE: 08028

FULL NAME AND RESIDENCE OF INVENTOR 4

LAST NAME FERNÁNDEZ LÓPEZ FIRST NAME Miquel Àngel MIDDLE NAME

CITY BARCELONA STATE OR FOREIGN COUNTRY SPAIN COUNTRY OF CITIZENSHIP SPAIN

POST OFFICE ADDRESS Poeta Cabanyes, 93 CITY Barcelona STATE OR COUNTRY Spain ZIP CODE 08004

FULL NAME AND RESIDENCE OF INVENTOR 5

LAST NAME ESPARDUCER BROCO FIRST NAME Araceli MIDDLE NAME

CITY Sant Pere de Ribes (Barcelona) STATE OR FOREIGN COUNTRY SPAIN COUNTRY OF CITIZENSHIP SPAIN

POST OFFICE ADDRESS Doctor Marañon, 32 CITY Sant Pere de Ribes (Barcelona) STATE OR COUNTRY Spain ZIP CODE 08810

FULL NAME AND RESIDENCE OF INVENTOR 6

LAST NAME SEGARRA RUBIK FIRST NAME Mercè MIDDLE NAME

CITY BARCELONA STATE OR FOREIGN COUNTRY SPAIN COUNTRY OF CITIZENSHIP SPAIN

POST OFFICE ADDRESS Alzinelles, 112 CITY Barcelona STATE OR COUNTRY Spain ZIP CODE 08014

FULL NAME AND RESIDENCE OF INVENTOR 7

LAST NAME CHIMENOS RIBERA FIRST NAME Josep Ma MIDDLE NAME

CITY BARCELONA STATE OR FOREIGN COUNTRY SPAIN COUNTRY OF CITIZENSHIP SPAIN

POST OFFICE ADDRESS Aristides Maillol, 11 CITY Barcelona STATE OR COUNTRY Spain ZIP CODE 08028

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

SIGNATURE OF INVENTOR 1: _____ DATED: 18.01.00

SIGNATURE OF INVENTOR 2: _____ DATED: 27-1-00

SIGNATURE OF INVENTOR 3: _____ DATED: 21-01-00

SIGNATURE OF INVENTOR 4: D. M. A Fernandez DATED: 21-01-00

SIGNATURE OF INVENTOR 5: ~~Oracci Espadura~~ DATED: 22-01-00

SIGNATURE OF INVENTOR 6: _____ DATED: 24-01-00

SIGNATURE OF INVENTOR 7: _____ DATED: 24-01-00